IS 5164 (1984): iso-borneol [PCD 18: Natural and Synthetic Fragrance Materials]

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(Reaffirmed 2002)

Indian Standard

SPECIFICATION FOR *iso*-BORNEOL

(First Revision)

UDC 665·574 : 547·599·4

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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November 1984

Indian Standard

SPECIFICATION FOR *iso*-BORNEOL

(First Revision)

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Indian Standard

SPECIFICATION FOR *iso*-BORNEOL

(First Revision)

0. FOREWORD

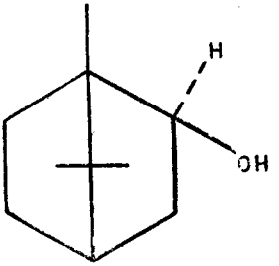
0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 16 August 1984, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969. The Sectional Committee responsible for its preparation felt that it should be revised with a view to bring it in line with the trade practices in perfumery technology, and also to align with the quality level of the material currently produced and sold in the country.

0.3 In this revision, the gas chromatographic method which is being progressively used in the country has been included and the wet method of analysis for determination of total alcohols has been deleted. Requirement for optical rotation has also been modified.

0.4 *iso*-Borneol ($C_{10}H_{18}O$) occurs rarely in nature and is reportedly extracted from *Abies sibirica* and *Chamaecyparis formosensis* both belonging to family Pinaceae. *iso*-Borneol is the structural isomer of borneol occurring in nature. It is a bicyclic terpene alcohol used primarily in perfumes, and to a much lesser degree in flavours. It may be prepared by the hydrolysis of *iso*-borneol acetate. It is an intermediate product in the manufacture of synthetic camphor from pinene, occurring in the oil of turpentine. Being comparatively inexpensive *iso*-borneol and its esters

are widely used in cosmetics, bath preparations, room sprays and in scenting of soaps. It is represented by the following structural formula:



iso-BORNEOL

(Molecular Mass 154·25)

0.5 In the preparation of this standard, considerable assistance has been derived from EOA No. 250, 1975 'Standard for *iso*-Borneol', published by the Essential Oil Association of USA, New York.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for *iso*-borneol.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 The material shall be tested olfactorily, for absence of earthy and burnt notes, as prescribed under **4** and **5** of IS : 2284-1963‡.

3.2 Solubility — The material shall be clearly soluble in three volumes of ethanol (70 percent by volume) when tested as prescribed under **8** of IS : 326-1968§.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Method for olfactory assessment of natural and synthetic perfumery materials.

§Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

3.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR *iso*-BORNEOL

(*Clauses* 3.3, 5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Colour and appearance	White crystalline or granular	IS : 326 (Part 2)-1980*	—
ii)	Odour	Strong camphoraceous	IS : 2284-1963†	—
iii)	Optical rotation [using 10 percent (*m/v*) solution in ethanol]	+1·5° to −1·5°	IS : 326 (Part 4)-1980*	—
iv)	Melting range, degree Celsius	209 to 214	16·9 of IS : 326-1968‡	—
v)	*iso*-Borneol content, percent by mass, *Min*	95	—	A
vi)	Total alcohols, percent by mass, *Min*	98	—	A

*Methods of sampling and test for natural and synthetic perfumery materials:

Part 2 Preliminary examination of perfumery materials and samples (*second revision*).

Part 4 Determination of optical rotation (*second revision*).

†Method for olfactory assessment of natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in tinplate containers or paper-lined wooden barrels.

4.1.1 The material shall be protected from light and stored in cool and dry place.

4.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Net mass of the material; and

d) Batch number.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by theprovisions of the Indian Standards Institution (Certification Mark) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

5.2 Number of Tests

5.2.1 *iso*-Borneol content shall be tested on each of the individual samples.

5.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity — The lot shall be declared as conforming to the requirements of the specification if **5.3.1** and **5.3.2** are satisfied.

5.3.1 For *iso*-borneol content, the mean ($\bar{X}$) and range (R) of test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of the test results}}$$

$$\text{Range } (R) = \text{Difference in the maximum and minimum of the test results}$$

The lot shall be deemed to have satisfied the requirement for this characteristic if the value of the expression $\bar{X} - 0{\cdot}6\,R$ is greater than or equal to the minimum limit for *iso*-borneol content given in Table 1.

5.3.2 All the test results on the composite sample meet the relevant specification requirements.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed under **3.1**, **3.2** and the appropriate references specified in col 4 and 5 of Table 1.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*), shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Items* (v) *and* (vi)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF *iso*-BORNEOL CONTENT

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph when it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for *iso*-borneol using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Sample	iso-*Borneol*
Column	
a) Material	Copper
b) Length	5·49 m
c) OD	0·635 cm
d) ID	0·476 cm
e) Stationary phase	FFAP†, 10 percent by mass
f) Solid support	Chromasorb WAW 60-80 mesh

*Specification for water for general laboratory use (*second revision*).
†Free fatty acid phase (FFAP) in carbowax 20M treated with nitrophathelic acid.

Sample	iso-*Borneol*
Carrier Gas	Hydrogen
Conditions	
a) Column temperature *iso*-thermal	150°C
b) Injection port temperature	200°C
c) Carrier gas flow rate	40 ml/min
d) Inlet pressure	3·5 kg/cm^2
Detector	
a) Type	Thermal conductively
b) Temperature	285°C
Recorder	
a) Span	1 mV
b) Chart speed	0·635 cm/min
Attenuation	4

NOTE — This analysis may also be accomplished with columns containing carbowax 20M, DE G.S (Diethylene glycol succinate).

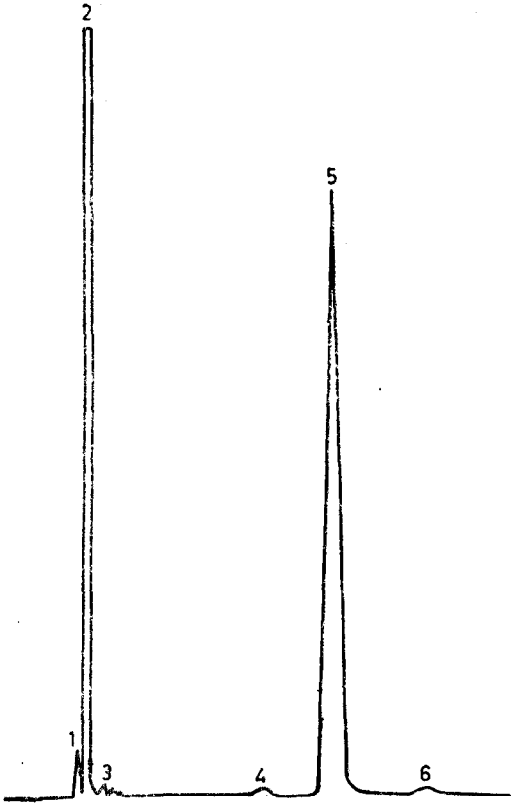
A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample (dissolved in the suitable solvent) at inject port when it is vaporized and well mixed with the carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

A-3. CALCULATION

A-3.1 Area Measurement (*See* Note 1) — Since normal peaks approximate a triangle the area is measured by multiplying the peak height times the width of half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.



1 — Air
2 — Solvent
3 — Tricyclene and camphene
4 — *iso*-Fenchyl alcohol
5 — *iso*-Borneol
6 — Borneol

FIG. 1 TYPICAL CHROMATOGRAM OF *iso*-BORNEOL

A-3.2 Area Normalization (*see* **Note 2**) — By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurements, namely, Triangulation, Disc Integrator and Electronic Digital Integrator if fixed with GLC machine would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is relative or indirect calibration.

(*Continued from page* 2)

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Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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